# SECTION 2 TECHNICAL PART

2.4 Confirmation analysis by GC/C/IRMS

2.4.1 GC/MS analysis

# TOPLEVEL PARAMETERS

```
lethod Information For: C:\MSDCHEM\1\METHODS\MAN_52.M
ethod Sections To Run:
 ( ) Save Copy of Method With Data
                        Pre-Run Cmd/Macro =
 ( ) MSTOP
  ( ) Instrument Control Pre-Run Cmd/Macro =
                       Pre-Run Cmd/Macro =
  ( ) Data Analysis
  (X) Data Acquisition
 (X) Data Analysis
                        Post-Run Cmd/Macro =
( ) MSTOP
( ) Instrument Control Post-Run Cmd/Macro = ( ) Data Analysis Post-Run Cmd/Macro =
1ethod Comments:
  This is the default method
                              END OF TOPLEVEL PARAMETERS
                              INSTRUMENT CONTROL PARAMETERS
                               ----
6890 GC METHOD
 OVEN
                                           Maximum temp: 325 'C
   Initial temp: 70 'C (On)
                                           Equilibration time: 0.50 min
   Initial time: 1.00 min
   Ramps:
         Rate Final temp Final time
      1 30.00 270 12.00
2 10.00 300 3.00
      3 0.0(Off)
    Post temp: 70 'C
Post time: 0.00 min
    Run time: 25.67 min
                                       BACK INLET (UNKNOWN)
 FRONT INLET (SPLIT/SPLITLESS)
    Mode: Splitless
    Initial temp: 280 'C (On)
Pressure: 145.0 kPa (On)
Purge flow: 20.0 mL/min
Purge time: 2.00 min
Total flow: 25.4 mL/min
Gas saver: Off
Gas type: Helium
                                         COLUMN 2
 COLUMN 1
                                            (not installed)
    Capillary Column
    Model Number: Agilent 19091s-433
    Max temperature: 325 'C
Nominal length: 30.0 m
    Nominal diameter: 250.00 um
    Nominal film thickness: 0.25 um
    Mode: constant pressure
     Pressure: 145.0 kPa
    Nominal initial flow: 2.3 mL/min
     Average velocity: 56 cm/sec
     Inlet: Front Inlet
     Outlet: MSD
     Outlet pressure: vacuum
                                                                    Page: 1
                             Mon Jul 24 14:19:20 2006
 Method: MAN_52.M
```

```
BACK DETECTOR ()
RONT DETECTOR ()
                                        SIGNAL 2
GINAL 1
                                           Data rate: 20 Hz
Data rate: 20 Hz
                                           Type: test plot
  Type: test plot
                                           Save Data: Off
Save Data: Off
                                           Zero: 0.0 (Off)
  Zero: 0.0 (Off)
                                           Range: 0
  Range: 0
                                           Fast Peaks: Off
  Fast Peaks: Off
Attenuation: 0
                                           Attenuation: 0
                                        COLUMN COMP 2
COLUMN COMP 1
                                           (No Detectors Installed)
  (No Detectors Installed)
THERMAL AUX 2
   Use: MSD Transfer Line Heater
  Description:
  Initial temp: 300 'C (On)
Initial time: 0.00 min
     # Rate Final temp Final time
        0.0(Off)
      1
                                       POST RUN
                                           Post Time: 0.00 min
IME TABLE
                                            Parameter & Setpoint
             Specifier
   Time
                             7673 Injector
     Front Injector:
                                      0
       Sample Washes
        Sample Pumps
                                    1.0 microliters
        Injection Volume
                                  10.0 microliters
        Syringe Size
        PostInj Solvent A Washes
                                      3
        PostInj Solvent B Washes
                                    3
3 seconds
        Viscosity Delay
                                   Fast
       Frunger Speed
PreInjection Dwell
        Plunger Speed
                                0.00 minutes
0.00 minutes
     PostInjection Dwell
    Back Injector:
 o parameters specified
 Column 1 Inventory Number : ?
 Column 2 Inventory Number :
                                 MS ACQUISITION PARAMETERS
 Seneral Information
 _____
                        : atune.u
: Scan
Tune File
 Acquistion Mode
 MS Information
 -- '-----
                         : 9.00 min
 Solvent Delay
                         : False
 EM Absolute
                        · · · · O
 EM Offset
                         : 1811.8
 Resulting EM Voltage
 [Scan Parameters]
                         : 50.0
Low Mass
                                                                    Page: 2
                            Mon Jul 24 14:19:20 2006
 Method: MAN_52.M
```

```
: 550.0
igh Mass
                       : 150
hreshold
                                  A/D Samples 4
Sample #
                    : 2
: 50.0
: 550.0
lot 2 high mass
[MSZones]
                        : 150 C maximum 200 C
S Quad
                        : 230 C maximum 250 C
S Source
                            END OF MS ACQUISITION PARAMETERS
                         END OF INSTRUMENT CONTROL PARAMETERS
                           DATA ANALYSIS PARAMETERS
Method Name: C:\MSDCHEM\1\METHODS\MAN_52.M
Percent Report Settings
| |----
Gort By: Signal
 output Destination
   Screen: No
Printer: Yes
             No
    File:
ntegration Events: AutoIntegrate
Generate Report During Run Method: No
 ignal Correlation Window: 0.020
 ualitative Report Settings
 Peak Location of Unknown: Apex
                     Minimum Quality
 ibrary to Search
C:\temp\IRMS.L
                       90
Integration Events: AutoIntegrate
 Report Type: Summary
 butput Destination
   Screen: No
Printer: Yes
     File:
             No
 Generate Report During Run Method: No
 Quantitative Report Settings
                                                                   Page: 3
                      Mon Jul 24 14:19:20 2006
Method: MAN_52.M
```

.sport Type: Summary putput Destination Screen: Yes Printer: No No File: enerate Report During Run Method: No alibration Last Updated: eference Window: 10.00 Percent on-Reference Window: 5.00 Percent Correlation Window: 0.02 minutes Default Multiplier: 1.00 efault Sample Concentration: 0.00 Compound Information \_\_\_\_\_ \*\*\* Empty Quantitation Database \*\*\* END OF DATA ANALYSIS PARAMETERS Mon Jul 24 14:19:20 2006

Method: MAN\_52.M

Mon Jul 24 14:19:20 2006

Page: 4

Method: MAN\_52.M

Mon Jul 24 14:19:20 2006

Page: 5

```
Sequence Name: C:\MSDChem\1\sequence\2006\Juil06\2307.S
          Comment:
         Operator: 49
        Data Path: D:\MSD22\JUIL06\2307\
Top
                     Pre-Seq Cmd:
 Instrument Control Pre-Seq Cmd:
 Data Analysis
                     Pre-Seq Cmd:
 Top
                     Post-Seq Cmd:
 Instrument Control Post-Seq Cmd:
 Data Analysis Post-Seq Cmd:
    Method Sections To Run
(X) Full Method
(X) Reprocessing Only

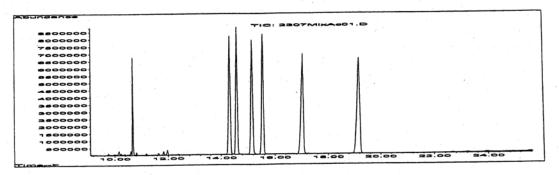
On A Barcode Mismatch
(X) Inject Anyway
( ) Don't Inject
                        Sample Name/Misc Info
    Line
  1) Calibration 1
                        2307MixAc01
      Datafile
                        MAN 52
      Method
                     2 blu1F3
                                 MAN_52
                                           Blu 1 F3
  2)
      Blank
3) Sample
                     3
      Datafile
                        17807474F3
                        MAN_52
4)
      Method
                     4 blu1F1
                                 MAN 52
                                         Blu 1 F1
     Blank
 5) Sample
                     5
                       17807474F1
      Datafile
                        MAN_52
      Method
                    6 blu1F2
                                  MAN_52 Blu 1 F2
  6)
     Blank
  7) Sample
                        17807474F2
      Datafile
                        MAN 52
      Method
  8) Sample
                        17807474F2b
      Datafile
      Method
                        MAN 52
                                                  Séquence vérifiée par : .....4.3
                                                                    ٧.
                                                  Remarques: .....
                                                                   -10
```

Last Modified: Sun Jul 23 10:17:10 2006

Page: 1

#### D:\Msd22\Juil06\2307\2307MixAc01.D

Data File Name 2307MixAc01.D
Data File Path D:\Msd22\Juil06\2307\
Operator 49
Date Acquired 7/23/2006 10:19
Acq. Method File MAN\_52.M
Sample Name Mix Ac 50
Vial Number 1
Misc Info Mix Acétate 001 50 ng injecté



Temps de rétention, temps de rétention relatif et target Signal (M1)

Name	Ret Time	Rel Ret Time	Target Signal	Target Response
5a Androstanol AC	10.69		258	10,892,705
Etiocholanolone AC	14.35	1.343	272	20,821,485
Androsterone AC	14.62	1.367	272	35,520,111
5b Androstan 3a 17b diol diAC	15.17	1.419	256	20,304,691
5a Androstan 3a 17b diol diAC	15.57	1.456	316	28,264,859
11 KetoEtiocholanolone AC	17.07	1.597	271	27,735,914
5b Pregnan 3a 20a diol diAC	19.20	1.796	284	35,625,886

#### M2 signal

Name	Q1 signal	Q1 Response	Q1 Ratio
5a Androstanol AC	243	11,496,221	105.5
Etiocholanolone AC	257	13,275,551	63.8
Androsterone AC	257	15,144,189	42.6
	316	18,349,659	90.4
5b Androstan 3a 17b diol diAC	241	16,541,690	58.5
5a Androstan 3a 17b diol diAC		23,029,104	83.0
11 KetoEtiocholanolone AC	191		
5b Pregnan 3a 20a diol diAC	269	20.053.051	56.3

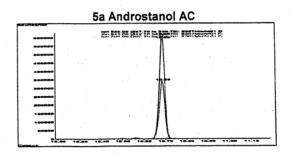
## M3 signal

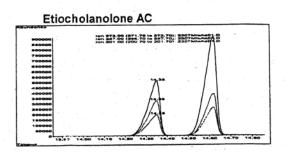
Page 1 of 2

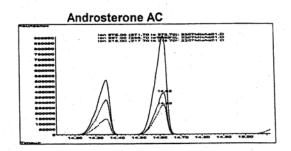
Name	Q2 signal	Q2 Response	Q2 Ratio
5a Androstanol AC	204	6,139,162	56.4
Etiocholanolone AC	201	7,984,811	38.3
Androsterone AC	218	10,868,162	30.6
	241	15,646,481	77.1
5b Androstan 3a 17b diol diAC	256	13,817,358	48.9
5a Androstan 3a 17b diol diAC	286	16,604,214	59.9
11 KetoEtiocholanolone AC		9,962,084	28.0
5b Pregnan 3a 20a diol diAC	344	9,962,064	20.0

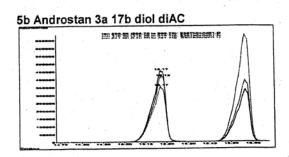
C:\MSDCHEM\CUSTRPT\MAN52.CRT

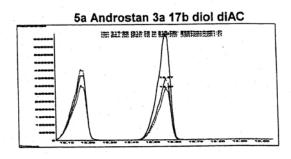
7/23/2006 11:33 AM

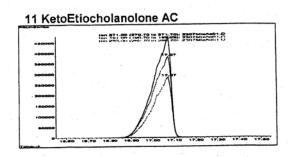


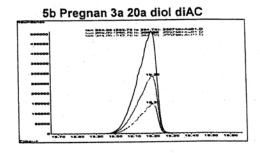










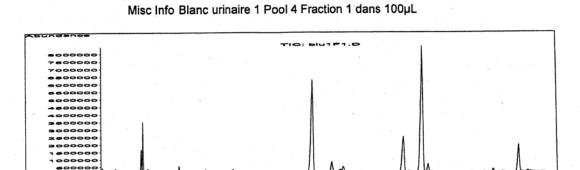


Page 2 of 2

7/23/2006 11:33 AM

#### D:\Msd22\Juil06\2307\blu1F1.D

Data File Name blu1F1.D
Data File Path D:\Msd22\Juil06\2307\
Operator 49
Date Acquired 7/23/2006 12:10
Acq. Method File MAN\_52.M
Sample Name Blu 1 F1
Vial Number 4



# Temps de rétention, temps de rétention relatif et target Signal (M1)

Name	Ret Time	Rel Ret Time	Target Signal	Target Response
5a Androstanol AC	10.68		258	5,234,507
Etiocholanolone AC	0.00	0.000	272	0
Androsterone AC	0.00	0.000	272	0
5b Androstan 3a 17b diol diAC	0.00	0.000	256	0
5a Androstan 3a 17b diol diAC	0.00	0.000	316	0
11 KetoEtiocholanolone AC	17.04	1.596	271	23,056,534
5b Pregnan 3a 20a diol diAC	0.00	0.000	284	0

#### M2 signal

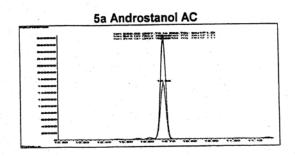
Name	Q1 signal	Q1 Response	Q1 Ratio
5a Androstanol AC	243	5,447,955	104.1
Etiocholanolone AC	257	0	0.0
Androsterone AC	257	. 0	0.0
5b Androstan 3a 17b diol diAC	316	0	0.0
5a Androstan 3a 17b diol diAC	241	0	0.0
11 KetoEtiocholanolone AC	191	19,045,346	82.6
5b Pregnan 3a 20a diol diAC	269	0	0.0

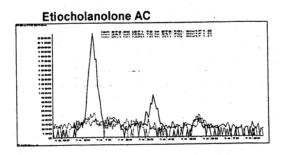
# M3 signal

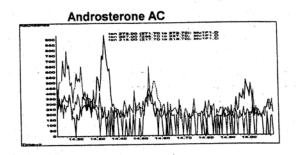
Name	Q2 signal	Q2 Response	Q2 Ratio
5a Androstanol AC	204	2,943,791	56.2
Etiocholanolone AC	201	0	0.0
Androsterone AC	218	0	0.0
5b Androstan 3a 17b diol diAC	241	0	0.0
5a Androstan 3a 17b diol diAC	256	0	0.0
11 KetoEtiocholanolone AC	286	13,914,444	60.3
5b Pregnan 3a 20a diol diAC	344	0	0.0

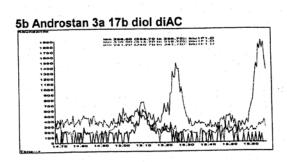
C:\MSDCHEM\CUSTRPT\MAN52.CRT

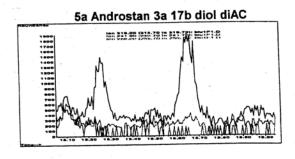
7/23/2006 12:55 PM

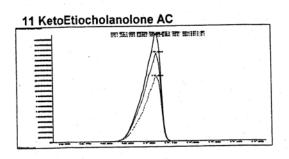


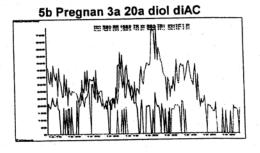












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7/23/2006 12:55 PM

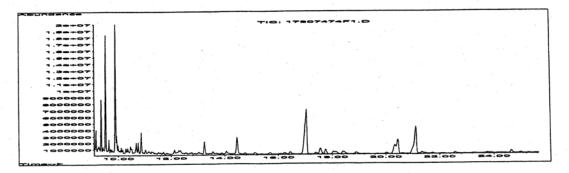
#### D:\Msd22\Juil06\2307\17807474F1.D

Data File Name 17807474F1.D
Data File Path D:\Msd22\Juil06\2307\
Operator 49
Date Acquired 7/23/2006 12:42
Acq. Method File MAN\_52.M

Sample Name 178/07 995474 F1

Vial Number 5

Misc Info 178/07 995474 Fraction 1 dans 100µL



# Temps de rétention, temps de rétention relatif et target Signal (M1)

Name	Ret Time	Rel Ret Time	Target Signal	Target Response
5a Androstanol AC	10.69		258	2,672,512
Etiocholanolone AC	0.00	0.000	272	0
Androsterone AC	0.00	0.000	272	0
5b Androstan 3a 17b diol diAC	0.00	0.000	256	0
5a Androstan 3a 17b diol diAC	0.00	0.000	316	0
11 KetoEtiocholanolone AC	17.05	1.596	271	27,520,882
5b Pregnan 3a 20a diol diAC	0.00	0.000	284	0

#### M2 signal

Name	Q1 signal	Q1 Response	Q1 Ratio
5a Androstanol AC	243	2,977,015	111.4
Etiocholanolone AC	257	0	0.0
Androsterone AC	257	0	0.0
5b Androstan 3a 17b diol diAC	316	0	0.0
5a Androstan 3a 17b diol diAC	241	0	0.0
11 KetoEtiocholanolone AC	191	22,323,512	81.1
5b Pregnan 3a 20a diol diAC	269	0	0.0

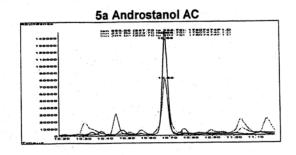
#### M3 signal

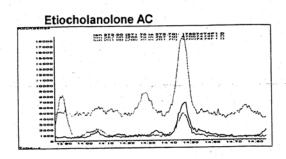
Name	Q2 signal	Q2 Response	Q2 Ratio
5a Androstanol AC	204	1,719,112	64.3
Etiocholanolone AC	201	0	0.0
Androsterone AC	218	0	0.0
5b Androstan 3a 17b diol diAC	241	0 ;	0.0
5a Androstan 3a 17b diol diAC	256	. 0	0.0
11 KetoEtiocholanolone AC	286	16,694,242	60.7
5b Pregnan 3a 20a diol diAC	344	0	0.0

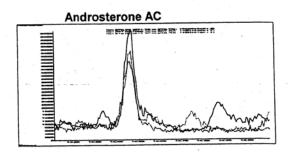
Page 1 of 2

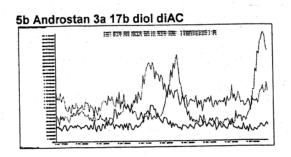
C:\MSDCHEM\CUSTRPT\MAN52.CRT

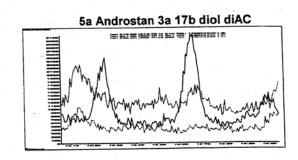
7/23/2006 1:14 PM

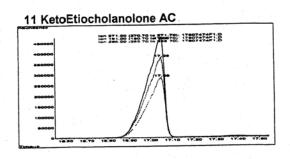


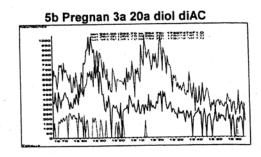












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C:\MSDCHEM\CUSTRPT\MAN52.CRT

7/23/2006 1:14 PM

#### D:\Msd22\Juil06\2307\blu1F2.D

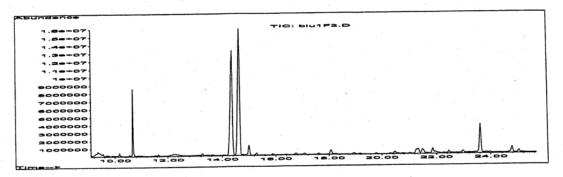
Data File Name blu1F2.D
Data File Path D:\Msd22\Juil06\2307\
Operator 49
Date Acquired 7/23/2006 13:15

Acq. Method File MAN\_52.M

Sample Name Blu 1 F2

Vial Number 6

Misc Info Blanc urinaire 1 Pool 4 Fraction 2 dans 400µL



# Temps de rétention, temps de rétention relatif et target Signal (M1)

Name	Ret Time	Rel Ret Time	Target Signal	Target Response
5a Androstanol AC	10.68		258	14,356,335
Etiocholanolone AC	14.39	1.347	272	46,218,242
Androsterone AC	14.66	1.373	272	92,159,652
,	0.00	0.000	256	0
5b Androstan 3a 17b diol diAC	0.00	0.000	316	0
5a Androstan 3a 17b diol diAC		0.000	271	0
11 KetoEtiocholanolone AC	0.00		284	n .
5b Pregnan 3a 20a diol diAC	0.00	0.000	204	

#### M2 signal

Name	Q1 signal	Q1 Response	Q1 Ratio
5a Androstanol AC	243	14,913,181	103.9
Etiocholanolone AC	257	29,244,244	63.3
Androsterone AC	257	39,385,747	42.7
		0	0.0
5b Androstan 3a 17b diol diAC	• . •		0.0
5a Androstan 3a 17b diol diAC		0	0.0
11 KetoEtiocholanolone AC	191	0	
5b Pregnan 3a 20a diol diAC	269	00	0.0

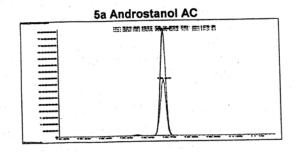
#### M3 signal

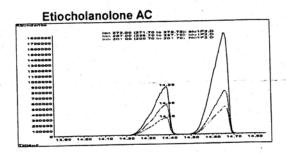
Page 1 of 2

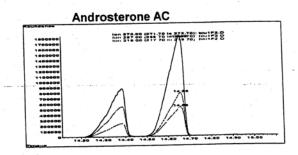
Name	Q2 signal	Q2 Response	Q2 Ratio
5a Androstanol AC	204	7,892,527	55.0
Etiocholanolone AC	201	16,995,418	36.8
Androsterone AC	218	26,814,361	29.1
5b Androstan 3a 17b diol diAC	241	0	0.0
5a Androstan 3a 17b diol diAC		0	0.0
	286	0 .	0.0
11 KetoEtiocholanolone AC	344	ň	0.0
5b Pregnan 3a 20a diol diAC	344		V.V.

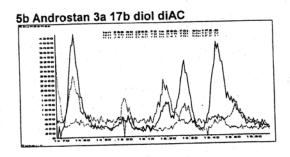
C:\MSDCHEM\CUSTRPT\MAN52.CRT

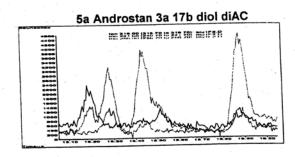
7/23/2006 2:23 PM

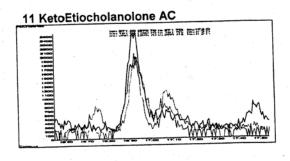


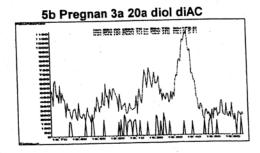












Page 2 of 2

7/23/2006 2:23 PM

#### D:\Msd22\Juil06\2307\17807474F2b.D

Data File Name 17807474F2b.D
Data File Path D:\Msd22\Juil06\2307\

Operator 49

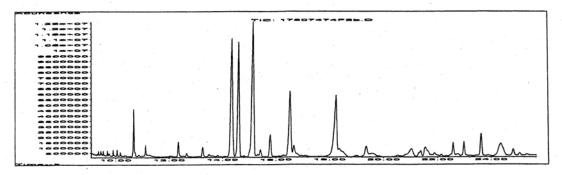
Date Acquired 7/23/2006 14:33

Acq. Method File MAN\_52.M

Sample Name 178/07 995474 F2

Vial Number 7

Misc Info 178/07 995474 Fraction 2 dans 400µL



Temps de rétention, temps de rétention relatif et target Signal (M1)

Name	Ret Time	Rel Ret Time	Target Signal	Target Response
5a Androstanol AC	10.69		258	6,790,329
Etiocholanolone AC	14.38	1.345	272	30,616,404
Androsterone AC	14.65	1.370	272	44,803,237
5b Androstan 3a 17b diol diAC	0.00	0.000	256	0
5a Androstan 3a 17b diol diAC	0.00	0.000	316	0 / .
11 KetoEtiocholanolone AC	0.00	0.000	271	0
5b Pregnan 3a 20a diol diAC	0.00	0.000	284	0

#### M2 signal

Name	Q1 signal	Q1 Response	Q1 Ratio
5a Androstanol AC	243	7,142,048	105.2
Etiocholanolone AC	257	19,396,055	63.4
Androsterone AC	257	19,255,218	43.0
5b Androstan 3a 17b diol diAC	316	0	0.0
5a Androstan 3a 17b diol diAC	241	0	0.0
11 KetoEtiocholanolone AC	191	0	0.0
5b Pregnan 3a 20a diol diAC	269	0	0.0

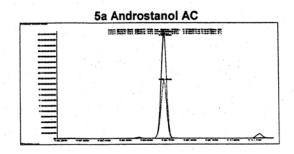
#### M3 signal

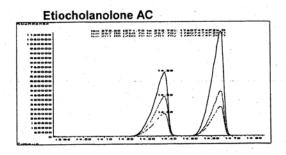
Name	Q2 signal	Q2 Response	Q2 Ratio
5a Androstanol AC	204	3,868,152	57.0
Etiocholanolone AC	201	11,568,610	37.8
Androsterone AC	218	13,516,338	30.2
5b Androstan 3a 17b diol diAC	241	0	0.0
5a Androstan 3a 17b diol diAC	256	0	0.0
11 KetoEtiocholanolone AC	286	0	0.0
5b Pregnan 3a 20a diol diAC	344	0 -	0.0

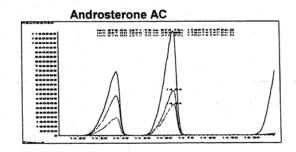
Page 1 of 2

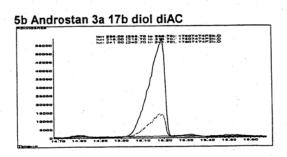
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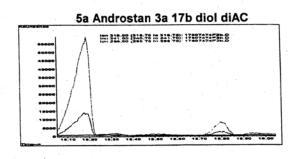
7/23/2006 3:01 PM

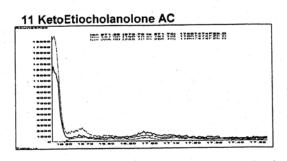


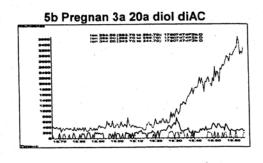












Page 2 of 2

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7/23/2006 3:01 PM

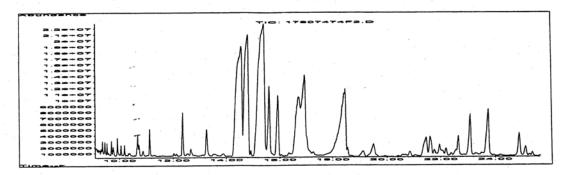
#### D:\Msd22\Juil06\2307\17807474F2.D

Data File Name 17807474F2.D
Data File Path D:\Msd22\Juil06\2307\
Operator 49
Date Acquired 7/23/2006 13:47
Acq. Method File MAN\_52.M

Sample Name 178/07 995474 F2

Vial Number 7

Misc Info 178/07 995474 Fraction 2 dans 100µL



# Temps de rétention, temps de rétention relatif et target Signal (M1)

Name	Ret Time	Rel Ret Time	Target Signal	Target Response
5a Androstanol AC	10.69		258	4,709,923
Etiocholanolone AC	14.58	1.365	272	169,848,822
Androsterone AC	14.84	1.388	272	213,976,918
5b Androstan 3a 17b diol diAC	0.00	0.000	256	0
5a Androstan 3a 17b diol diAC	0.00	0.000	316	0
11 KetoEtiocholanolone AC	0.00	0.000	271	0
5b Pregnan 3a 20a diol diAC	0.00	0.000	284	0

#### M2 signal

Name	Q1 signal	Q1 Response	Q1 Ratio
5a Androstanol AC	243	5,011,709	106.4
Etiocholanolone AC	257	104,358,276	61.4
Androsterone AC	257	91,230,541	42.6
5b Androstan 3a 17b diol diAC	316	0	0.0
5a Androstan 3a 17b diol diAC	241	0	0.0
11 KetoEtiocholanolone AC	191	0	0.0
5b Pregnan 3a 20a diol diAC	269	0	0.0

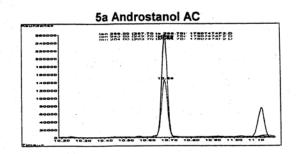
## M3 signal

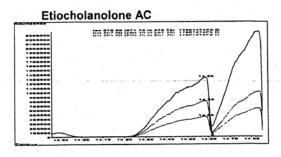
Name	Q2 signal	Q2 Response	Q2 Ratio
5a Androstanol AC	204	2,896,716	61.5
Etiocholanolone AC	201	59,514,197	35.0
Androsterone AC	218	61,028,102	28.5
5b Androstan 3a 17b diol diAC	241	0	0.0
5a Androstan 3a 17b diol diAC		0	0.0
11 KetoEtiocholanolone AC	286	0	0.0
5b Pregnan 3a 20a diol diAC	344	0	0.0

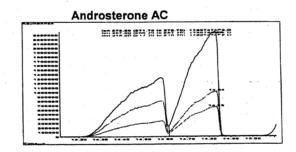
Page 1 of 2

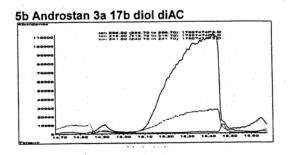
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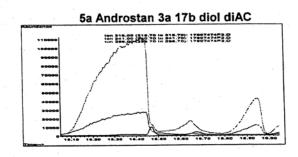
7/23/2006 2:22 PM

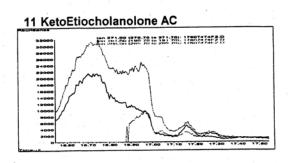


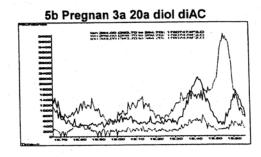












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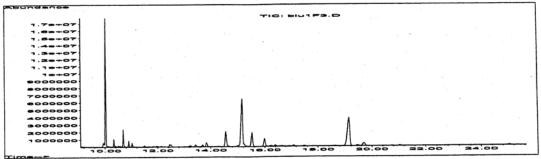
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7/23/2006 2:22 PM

#### D:\Msd22\Juil06\2307\blu1F3.D

Data File Name blu1F3.D
Data File Path D:\Msd22\Juil06\2307\
Operator 49
Date Acquired 7/23/2006 11:00
Acq. Method File MAN\_52.M
Sample Name Blu 1 F3
Vial Number 2

Misc Info Blanc urinaire 1 Pool 4 Fraction 3 dans 100μL



# Temps de rétention, temps de rétention relatif et target Signal (M1)

Name	Ret Time	Rel Ret Time	Target Signal	Target Response
5a Androstanol AC	10.68		258	3,476,361
Etiocholanolone AC	0.00	0.000	272	0
Androsterone AC	0.00	0.000	272	0
5b Androstan 3a 17b diol diAC	15.17	1.420	256	15,597,241
5a Androstan 3a 17b diol diAC	15.51	1.452	316	5,215,670
11 KetoEtiocholanolone AC	0.00	0.000	271	0
5b Pregnan 3a 20a diol diAC	19.14	1.792	284	16,549,438

### M2 signal

Name	Q1 signal	Q1 Response	Q1 Ratio
5a Androstanol AC	243	3,650,999	105.0
Etiocholanolone AC	257	0	0.0
Androsterone AC	257	. 0	0.0
5b Androstan 3a 17b diol diAC	316	14,170,551	90.9
5a Androstan 3a 17b diol diAC	241	3,187,095	61.1
11 KetoEtiocholanolone AC	191	0	0.0
5b Pregnan 3a 20a diol diAC	269	9,365,521	56.6

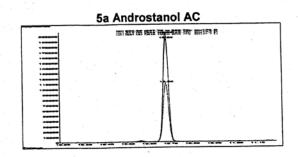
#### M3 signal

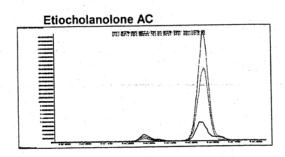
Page 1 of 2

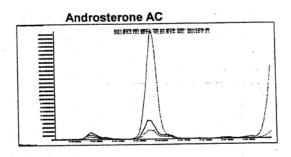
Name	Q2 signal	Q2 Response	Q2 Ratio
5a Androstanol AC	204	1,954,169	56.2
Etiocholanolone AC	201	0	0.0
Androsterone AC	218	0	0.0
5b Androstan 3a 17b diol diAC	241	12,280,836	78.7
5a Androstan 3a 17b diol diAC	256	2,634,418	50.5
11 KetoEtiocholanolone AC	286	0	0.0
5b Pregnan 3a 20a diol diAC	344	4,508,073	27.2

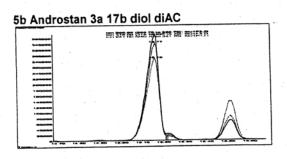
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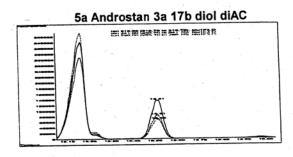
7/23/2006 11:30 AM

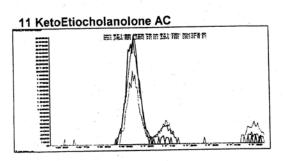


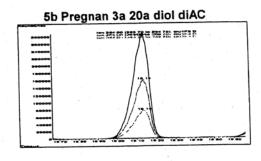










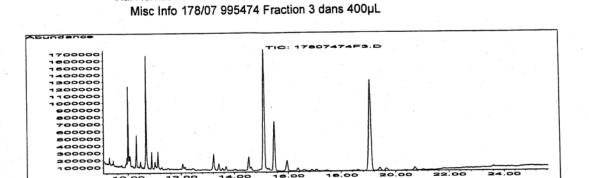


Page 2 of 2

7/23/2006 11:30 AM

#### D:\Msd22\Juil06\2307\17807474F3.D

Data File Name 17807474F3.D
Data File Path D:\Msd22\Juil06\2307\
Operator 49
Date Acquired 7/23/2006 11:33
Acq. Method File MAN\_52.M
Sample Name 178/07 995474 F3
Vial Number 3



# Temps de rétention, temps de rétention relatif et target Signal (M1)

Name	Ret Time	Rel Ret Time	Target Signal	Target Response
5a Androstanol AC	10.67	,	258	2,492,729
Etiocholanolone AC	0.00	0.000	272	0
Androsterone AC	0.00	0.000	272	0
5b Androstan 3a 17b diol diAC	15.10	1.415	256	3,658,105
5a Androstan 3a 17b diol diAC	15.48	1.450	316	1,968,201
11 KetoEtiocholanolone AC	0.00	0.000	271	0
5b Pregnan 3a 20a diol diAC	19.06	1.786	284	4,951,688

## M2 signal

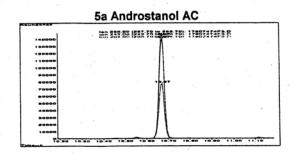
Name	Q1 signal	Q1 Response	Q1 Ratio
5a Androstanol AC	243	2,615,044	104.9
Etiocholanolone AC	257	0	0.0
Androsterone AC	257	0	0.0
5b Androstan 3a 17b diol diAC	316	3,242,469	88.6
5a Androstan 3a 17b diol diAC	241	1,199,926	61.0
	191	0	0.0
11 KetoEtiocholanolone AC	269	2.833.947	57.2
5b Pregnan 3a 20a diol diAC	209	2.000.047	

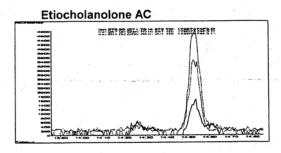
#### M3 signal

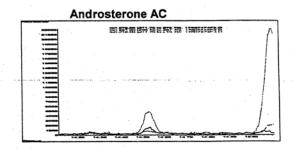
Q2 signal	Q2 Response	Q2 Ratio
204	1,395,444	56.0
201	0	0.0
218	0	0.0
241	2,877,543	78.7
		50.1
	0	0.0
	1 343 347	27.1
	204 201	204 1,395,444 201 0 218 0 241 2,877,543 256 985,712 286 0

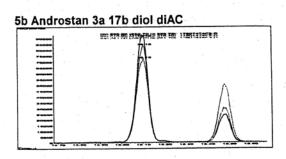
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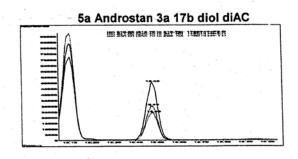
7/23/2006 12:00 PM

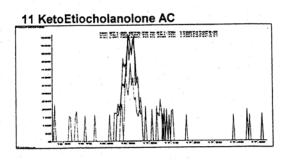


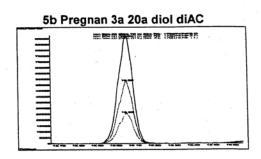












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7/23/2006 12:00 PM

LNDD

# **ENREGISTREMENT**

Codification: E-CC-11

Version: B

Date: 08/03/2006

1/1

# VERIFICATION DES PERFORMANCES INSTRUMENTALES EN CG/SM (screening et confirmation)

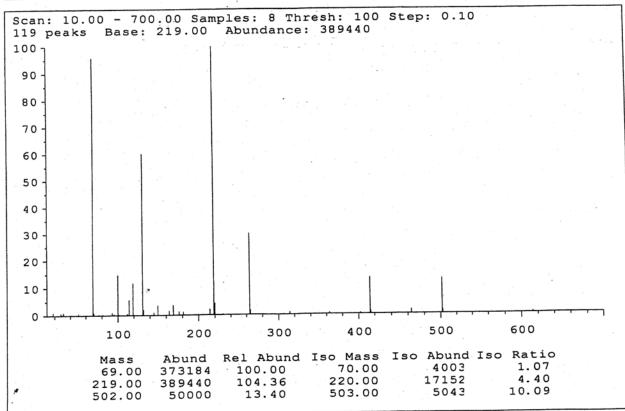
Numéro	o d'identification de l'appareil : الله هنگ	
	Date: 23/07/06	
1 - Source d'ic	onisation	Oni Non
MSD Polaris	Autotune: Ion 69 ou 219 majoritaire Autotune: Abondance de l'ion 502 > 3% Repeller < 35 Ion time > 2 ms	Oui Non
Observations:		
2 - Etanchéité	du système	Oui Non
MSD	18/69 ( H2O), 28/69 (N2), 32/69 (O2), 44/69 (CO2) < 10%	α I
Polaris	Air et eau : Intensité ion 19 < intensité ion 1	8
Observations		
3 - Sensibilité		Oui Non
Screening Conf	Recal / Mix conforme  TP conforme - Fichier: 2307 Nic Acol  TP conforme - Fichier:  TP conforme - Fichier:  TP conforme - Fichier:  TP conforme - Fichier:	α
Observations		
Code opérate	ur et paraphe : 49	

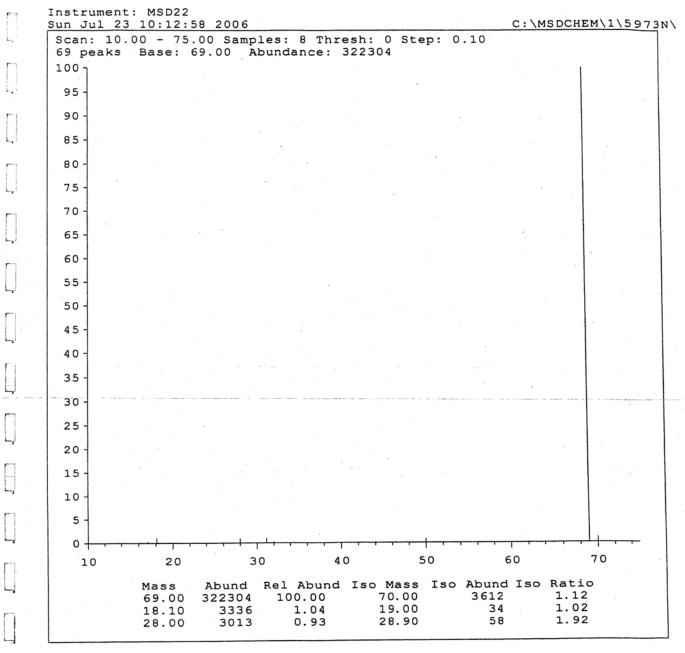
Cet enregistrement est à archiver dans le classeur C-MA-Ech de l'appareil

sun Jul 23 10:11:55 2006
C:\MSDCHEM\1\5973N\ATUNE.U

Instrument: MSD22

			— т						
Mass Ab	69.00 421715 0.61		19.00 37153 0.61	Mass Ab Pw50	502.00 54028 0.62	Ion Pol	POS	MassGain MassOffs	267 -10
Pw50	0.61	PWSU	0.61	FWJU	, 0,02	Emission	34.6	AmuGain	2179
	1	l . A			Λ 1	ElEnrgy	69.9	AmuOffs	130
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	11		-		11 1	Repeller	24.26		
						IonFcus		HEDEnab	ON
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		1 . 11			1 .	EntOffs	17.57		
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Current Params used: ATUNE.U

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Relative abundances:

18/69 = 1.04 Water%

28/69 = 0.93 Nitrogen%

32/69 = 0.28 Oxygen%

44/69 = 0.17 Carbon Dioxide%

28/18 = 90.32 Nitrogen/Water%
```

LNDD  FICHE D'ANALYSE / RESULTA			:	ENREGISTREMENT				1/3	E-FCR-09 A 31/01/2006 1/3	
FICHE D'AN	NALYSE / R	ESULTAT	ANALYS	SE QUALI'	TATIVE (	GC/MS POU	R CONFI	RMATION	GC/C/IR	MS
Echantillon:	·	178/07 9	005474							
Echantinon .	L	170/07	773474							
Tolérances fixées p	ar l'AMA ( d	locument · 1	WADA Tec	chnical Doc	ument - TI	)2003IDCB )				
r orerances fixees p	a min (	iocument.	WADA ICC	omnear Doc	ument - 11	2003IDCK )				
l'olérances sur le tr	et le trr :+/	-1% ou +/-	0,2 min (pr	endre la plu	us faible de	s deux)				
Pour les abondance	s relatives	> 50%	il est	admis	+/- 10% (	en absolu ) de	variation			
. , , ,		25 <ab< 50<="" td=""><td></td><td></td><td>+/- 20% (</td><td></td><td>· variation</td><td></td><td></td><td></td></ab<>			+/- 20% (		· variation			
		< 25%			+/- 5% (er					
.**	, ,									
						_				
Standard Interne:										*
Standard Interne:	М	(ix	Fract	ion F1	Fract	ion F2	Fracti	on F3	·	
	<b>M</b>			ion F1		ion F2	Fraction 10.0			
Standard Interne:  Tr (min)  Fichier		.69	10		10			67		
Tr (min)	10. 2307M	.69 ixAc01	10	.69 /474F1	10	.69	10.4 178074	67		
Tr (min) Fichier	10. 2307M	.69 ixAc01	10 17807	.69 /474F1	10	.69 474f2b	10.0 178074	67 174F3		
Tr (min) Fichier	10. 2307M	.69 ixAc01	10 17807 étiocholano	.69 /474F1	10 17807	.69 474f2b	10.0 178074	67 174F3 7807474F1	M2 (%)	М3 (1
Tr (min) Fichier	10. 2307M etérisée :	69 ixAc01 11 Kétoo	10 17807 étiocholano Mix	.69 7474F1 clone AC M2 (%) 83	10 17807	.69 474f2b Fichier :	10.4 178074	67 174F3 7807474F1		
Tr (min) Fichier  Substance carac	10. 2307M  etérisée :  Tr (min) 17.07 16.90	11 Kétod Trr 1.597	10 17807 étiocholano Mix M1 (%)	M2 (%) 83 73	M3 (%) 59.9 49.9	.69 474f2b Fichier : [	10.4 178074 1 1 Е	7807474F1 Chantillon M1 (%)	M2 (%)	M3 (9 60.7
Tr (min) Fichier  Substance carac	10. 2307M  ctérisée :  Tr (min) 17.07	69 ixAc01  11 Kétoo  Trr 1.597	10 17807 étiocholano Mix M1 (%)	.69 7474F1 clone AC M2 (%) 83	10 17807 M3 (%) 59.9	.69 474f2b Fichier : [	10.4 178074 1 1 Е	7807474F1 Chantillon M1 (%)	M2 (%)	
Tr (min) Fichier  Substance carac  Tolérance basse Tolérance haute	10. 2307M  etérisée :  Tr (min) 17.07 16.90 17.24	11 Kétod Trr 1.597	10 17807 étiocholano Mix M1 (%)	M2 (%) 83 73 93	M3 (%) 59.9 49.9 69.9	Fichier : Tr (min) 17.05	10.4 178074 1 1 Е	7807474F1 Chantillon M1 (%)	M2 (%)	
Tr (min) Fichier  Substance carac  Tolérance basse Tolérance haute  Concordance des T	10. 2307M  etérisée :  Tr (min) 17.07 16.90 17.24	11 Kétod Trr 1.597	10 17807 étiocholano Mix M1 (%)	M2 (%) 83 73	M3 (%) 59.9 49.9 69.9	.69 474f2b Fichier : [	10.4 178074 1 1 Е	7807474F1 Chantillon M1 (%)	M2 (%)	
Tr (min) Fichier  Substance carac  Tolérance basse Tolérance haute  Concordance des T  Concordance des T	10. 2307M  etérisée :  Tr (min) 17.07 16.90 17.24  r :	11 Kétod  Trr 1.597 1.581 1.613	10 17807 étiocholano Mix M1 (%) 100	M2 (%) 83 73 93	M3 (%) 59.9 49.9 69.9	Fichier : Tr (min) 17.05	10.4 178074 1 1 Е	7807474F1 Chantillon M1 (%)	M2 (%)	
Tr (min) Fichier  Substance carac	10. 2307M  etérisée :  Tr (min) 17.07 16.90 17.24  r :	11 Kétod  Trr 1.597 1.581 1.613	100 17807	M2 (%) 83 73 93 oui oui	M3 (%) 59.9 49.9 69.9  X X	Fichier : Tr (min) 17.05	10.4 178074 1 1 Е	7807474F1 Chantillon M1 (%)	M2 (%)	
Tr (min) Fichier  Substance carac  Tolérance basse Tolérance haute  Concordance des T  Concordance des T	10. 2307M  etérisée :  Tr (min) 17.07 16.90 17.24  r :	11 Kétod  Trr 1.597 1.581 1.613	100 17807	M2 (%) 83 73 93 oui oui	M3 (%) 59.9 49.9 69.9  X X	Fichier : Tr (min) 17.05	10.4 178074 1 1 Е	7807474F1 Chantillon M1 (%)	M2 (%)	

				Mix				I	Echantillon		
		Tr (min)	Trr	M1 (%)	M2 (%)	M3 (%)	Tr (min)	Trr	M1 (%)	M2 (%)	M3 (%)
		14.35	1.343	100	63.8	42.6	14.38	1.345	100	63.4	37.8
	Tolérance basse	14.21	1.330		53.8	34.08					
1	Tolérance haute	14.49	1.356		73.8	51.12					
					,						

Concordance des Tr :	oui	Х	non	
Concordance des Trr:	oui	Х	non	
Concordance globale des abondances relatives :	oui	Х	non	

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# **ENREGISTREMENT**

Codification:

E-FCR-09

Version:
Date:

31/01/2006

2/3

FICHE D'ANALYSE / RESULTAT ANALYSE QUALITATIVE GC/MS POUR CONFIRMATION GC/C/IRMS

Substance carac	térisée :	And	rostérone A	.C		Fichier:	17	807474F2b		
			Mix	-	T	Echantillon				
	Tr (min)	Trr	M1 (%)	M2 (%)	M3 (%)	Tr (min)	Тп	M1 (%)	M2 (%)	M3 (%
	14.62	1.367	100	42.6	30.6	14.65	1.370	100	43	30.2
Tolérance basse	14.47	1.353		34.08	24.48					
Tolérance haute	14.77	1.381		51.12	36.72					
,						_		i		
Concordance des T				oui	X	non				
Concordance des T			.'	oui	X	non				
Concordance globa	le des abond	ances relati	ves:	oui [	X	non [		j		
• .										
										,
Substance carac	ctérisée :	5b Andros	tane-3a,17b-	diol diAC		Fichier:	. 1	7807474F3		
			- 77:			<u> </u>	- · · ·	Echantillon		
1			Mix	10 (0/)	M3 (%)	Tr (min)	Trr	M1 (%)	M2 (%)	M3 (9
	Tr (min)	Trr	M1 (%)	M2 (%)	77.1	15.1	1.415	100	88.6	78.7
m 1/ 1	15.17	1.419	100	90.4 80.4	67.1	13.1	1.415	100	33.0	
Tolérance basse	15.02 15.32	1.405		100.4	87.1					
Tolérance haute	13.32	1.433	and a six top see your of	100.1	07.12					
				:	Х	non [		1		
T ach academic dea	tara di mana									
Concordance des T				oui		1		1 '		
Concordance des T	îrr:	lances relat	ives	oui	X	non		-		
	îrr:	lances relat	ives :			1		1		
Concordance des T	îrr:	lances relat	ives :	oui	X	non				
Concordance des T	îrr:	lances relat	ives :	oui	X	non non				
Concordance des T	rr: ale des abono		ives :	oui oui	X	non	1,000	17807474F	3	]
Concordance des T Concordance globa	rr: ale des abono		stane-3a,17b	oui oui	X	non non				]
Concordance des T Concordance globa	Tr: ale des abono ctérisée :	5a Andro	stane-3a,17b Mix	oui oui	x x	non non Fichier:		17807474F Echantillon M1 (%)		] M3 (
Concordance des T Concordance globa	Tr (min)	5a Andro	stane-3a,17b Mix M1 (%)	oui oui diol diAC	X	non non		Echantillon		
Concordance des T Concordance globa Substance cara	Tr (min) 15.57	5a Andro  Trr  1.456	stane-3a,17b Mix	oui oui	X X	non non Fichier:	Тпт	Echantillon M1 (%)	M2 (%)	
Concordance des T Concordance globa  Substance cara  Tolérance basse	Tr (min)	5a Andro	stane-3a,17b Mix M1 (%)	oui oui diol diAC	X X X M3 (%) 48.9	non non Fichier:	Тпт	Echantillon M1 (%)	M2 (%)	
Concordance des T Concordance globa Substance cara	Tr (min) 15.57 15.41	5a Andro  Trr 1.456 1.441	stane-3a,17b Mix M1 (%)	oui oui diol diAC M2 (%) 58.5 48.5	X X X M3 (%) 48.9 39.12 58.68	richier: Tr (min) 15.48	Тпт	Echantillon M1 (%)	M2 (%)	
Concordance des T Concordance globa  Substance cara  Tolérance basse	Tr (min) 15.57 15.41 15.73	5a Andro  Trr 1.456 1.441	stane-3a,17b Mix M1 (%)	oui oui diol diAC M2 (%) 58.5 48.5 68.5	X X X M3 (%) 48.9 39.12 58.68	richier:  Tr (min) 15.48	Тпт	Echantillon M1 (%)	M2 (%)	
Concordance des T Concordance globa  Substance cara  Tolérance basse Tolérance haute  Concordance des T Concordance des T	Tr : ale des abono ctérisée :  Tr (min) 15.57 15.41 15.73  Tr :	5a Andro  Tπ 1.456 1.441 1.471	Mix M1 (%) 100	oui oui diol diAC M2 (%) 58.5 48.5 68.5	X X X 39.12 58.68 X X	richier: Tr (min) 15.48 non non	Тпт	Echantillon M1 (%)	M2 (%)	_
Concordance des T Concordance globa  Substance cara  Tolérance basse Tolérance haute  Concordance des T	Tr : ale des abono ctérisée :  Tr (min) 15.57 15.41 15.73  Tr :	5a Andro  Tπ 1.456 1.441 1.471	Mix M1 (%) 100	oui oui diol diAC M2 (%) 58.5 48.5 68.5	X X X M3 (%) 48.9 39.12 58.68	richier:  Tr (min) 15.48	Тпт	Echantillon M1 (%)	M2 (%)	_
Concordance des T Concordance globa  Substance cara  Tolérance basse Tolérance haute  Concordance des T Concordance des T	Tr : ale des abono ctérisée :  Tr (min) 15.57 15.41 15.73  Tr :	5a Andro  Tπ 1.456 1.441 1.471	Mix M1 (%) 100	oui oui diol diAC M2 (%) 58.5 48.5 68.5	X X X 39.12 58.68 X X	richier: Tr (min) 15.48 non non	Тпт	Echantillon M1 (%)	M2 (%)	_
Concordance des T Concordance globa  Substance cara  Tolérance basse Tolérance haute  Concordance des T Concordance des T	Tr : ale des abono ctérisée :  Tr (min) 15.57 15.41 15.73  Tr :	5a Andro  Tπ 1.456 1.441 1.471	Mix M1 (%) 100	oui oui diol diAC M2 (%) 58.5 48.5 68.5	X X X 39.12 58.68 X X	richier: Tr (min) 15.48 non non	Тпт	Echantillon M1 (%)	M2 (%)	_
Concordance des T Concordance globa  Substance cara  Tolérance basse Tolérance haute  Concordance des T Concordance des T Concordance glob	Tr : ale des abond ctérisée :  Tr (min) 15.57 15.41 15.73  Tr : Trr : ale des abon	5a Andro  Trr  1.456  1.441  1.471  dances rela	Mix M1 (%) 100	oui oui diol diAC M2 (%) 58.5 48.5 0ui oui	X X X 39.12 58.68 X X	richier:  Tr (min) 15.48  non non non	Тпт	Echantillon M1 (%)	M2 (%)	_
Concordance des T Concordance globa  Substance cara  Tolérance basse Tolérance haute  Concordance des T Concordance des T	Tr : ale des abond ctérisée :  Tr (min) 15.57 15.41 15.73  Tr : Trr : ale des abon	5a Andro  Trr  1.456  1.441  1.471  dances rela	Mix M1 (%) 100	oui oui diol diAC M2 (%) 58.5 48.5 0ui oui	X X X 39.12 58.68 X X	richier: Tr (min) 15.48 non non	Тпт	Echantillon M1 (%) 100	M2 (%) 61	_
Concordance des T Concordance globa  Substance cara  Tolérance basse Tolérance haute  Concordance des T Concordance des T Concordance glob	Tr : ale des abond ctérisée :  Tr (min) 15.57 15.41 15.73  Tr : Trr : ale des abon	5a Andro  Trr  1.456  1.441  1.471  dances rela	Mix M1 (%) 100	oui oui diol diAC M2 (%) 58.5 48.5 0ui oui	X X X 39.12 58.68 X X	richier:  Tr (min) 15.48  non non non	Тпт	Echantillon M1 (%) 100  17807474	M2 (%) 61	50.
Concordance des T Concordance globa  Substance cara  Tolérance basse Tolérance haute  Concordance des T Concordance des T Concordance glob	Tr : ale des abond  ctérisée :  Tr (min) 15.57 15.41 15.73  Tr : ale des abon  actérisée :	5a Andro  Trr  1.456  1.441  1.471  dances rela	Mix M1 (%) 100  tives:	oui oui diol diAC M2 (%) 58.5 48.5 0ui oui	X X X 39.12 58.68 X X X	richier:  Tr (min) 15.48  non non non Fichier:	Тп 1.450	Echantillon M1 (%) 100  178074741  Echantillo M1 (%)	M2 (%) 61	50.
Concordance des T Concordance globa  Substance cara  Tolérance basse Tolérance haute  Concordance des T Concordance des T Concordance glob	Tr (min) 15.57 15.41 15.73  Tr : ale des abon  Tr (min)	5a Andro  Trr 1.456 1.441 1.471  dances rela	Mix M1 (%) 100 tives:	oui oui diol diAC  M2 (%) 58.5 48.5 68.5 oui oui oui	X X X 39.12 58.68 X X X	richier:  Tr (min) 15.48  non non non Fichier:	Тп 1.450	Echantillon M1 (%) 100  178074741  Echantillo M1 (%)	M2 (%) 61	50.
Concordance des T Concordance globa  Substance cara  Tolérance basse Tolérance haute  Concordance des T Concordance glob  Substance cara	Tr : ale des abond  ctérisée :  Tr (min) 15.57 15.41 15.73  Tr : ale des abon  actérisée :  Tr (min) 19.2	5a Andro  Trr 1.456 1.441 1.471  dances rela  5b Prég	Mix M1 (%) 100 tives:	oui oui diol diAC  M2 (%) 58.5 48.5 68.5  oui oui oui diol diAC	X X X 39.12 58.68 X X X	richier:  Tr (min) 15.48  non non non Tr (min)	Тп 1.450	Echantillon M1 (%) 100  178074741  Echantillo M1 (%)	M2 (%) 61	50.
Concordance des T Concordance globa  Substance cara  Tolérance basse Tolérance haute  Concordance des T Concordance glob  Substance cara	Tr (min) 15.57 15.41 15.73  Tr : ale des abond  Tr (min) 19.2 19.01	5a Andro  Trr 1.456 1.441 1.471  dances rela  5b Prég  Trr 1.796	Mix M1 (%) 100 tives:	oui oui oui M2 (%) 58.5 48.5 68.5 oui oui oui diol diAC	X X X 39.12 58.68 X X X X	richier:  Tr (min) 15.48  non non non Tr (min)	Тп 1.450	Echantillon M1 (%) 100  178074741  Echantillo M1 (%)	M2 (%) 61	50 M3
Concordance des T Concordance globa  Substance cara  Tolérance basse Tolérance haute  Concordance des T Concordance glob  Substance cara	Tr : ale des abond  ctérisée :  Tr (min) 15.57 15.41 15.73  Tr : ale des abon  actérisée :  Tr (min) 19.2	5a Andro  Trr 1.456 1.441 1.471  dances rela  5b Prég  Trr 1.796 1.778	Mix M1 (%) 100 tives:	oui oui oui M2 (%) 58.5 48.5 68.5 oui oui oui M2 (%) 56.3 46.3	X X X 48.9 39.12 58.68 X X X 3.10 2.10 2.10 2.10 2.10 2.10 2.10 3.10 2.10 3.10 3.10 3.10 3.10 3.10 3.10 3.10 3	richier:  Tr (min) 15.48  non non non Tr (min)	Тп 1.450	Echantillon M1 (%) 100  178074741  Echantillo M1 (%)	M2 (%) 61	50 M3
Concordance des T Concordance globa  Substance cara  Tolérance basse Tolérance haute  Concordance des T Concordance glob  Substance cara	Tr : ale des abond  ctérisée :  Tr (min) 15.57 15.41 15.73  Tr : ale des abond  actérisée :  Tr (min) 19.2 19.01 19.39	5a Andro  Trr 1.456 1.441 1.471  dances rela  5b Prég  Trr 1.796 1.778	Mix M1 (%) 100 tives:	oui oui oui M2 (%) 58.5 48.5 68.5 oui oui oui M2 (%) 56.3 46.3	X X X 39.12 58.68 X X X X	richier:  Tr (min) 15.48  non non non Tr (min)	Тп 1.450	Echantillon M1 (%) 100  178074741  Echantillo M1 (%)	M2 (%) 61	50 M3

oui

Concordance globale des abondances relatives :

USADA 0150

non

LNDD		ENRE	EGISTREM	MENT	Codification: E-FCR-09 Version: A Date: 31/01/2006 3/3					
FICHE D'AI	NALYSE / R	ESULTA?	Γ ANALYS	E QUALI	TATIVE	GC/MS PO	UR CONF	IRMATIO	N GC/C/II	RMS
Substance carac	ctérisée : [				1	Fichier:	ī.			
					1 .	, ,				
•			Mix				]	Echantillon		
	Tr (min)	Trr	M1 (%)	M2 (%)	M3 (%)	Tr (min)	Trr	M1 (%)	M2 (%)	M3 (9
			100				<u> </u>	100		
Tolérance basse										
Tolérance haute						, , ,				
Concordance des T	<b>.</b> .			oui		non	r i	1		
Concordance des T				oui		non		1 .		
Concordance globa		ances relat	ives:	oui		non				
						•				
			٠.							
					1					, .
Substance cara	ctérisée :					Fichier:				]
		<del></del>	Mix			<u> </u>		Echantillon		
	Tr (min)	Trr	M1 (%)	M2 (%)	M3 (%)	Tr (min)	Trr	M1 (%)	M2 (%)	M3 (9
	II (IIIII)		100	1,12 (75)	1125 (75)		1	100	1	
Tolérance basse										
Tolérance haute										
						1		· ·		
Concordance des T				oui oui		non		-		;
Concordance des T					•					
Canaardanaa alaha	le des aband	ances relat	ivec :			non				
Concordance globa	ale des abond	ances relat	ives:	oui		non		<u>.</u>		
Concordance globa	ale des abond	ances relat	ives:			-				
Concordance globa	ale des abond	ances relat	ives:			-		<b>]</b>		
Concordance globa  Paraphe et code op		ances relat			1	-				
		ances relat	ives :			-				
Paraphe et code op	érateur :					-				
	érateur :					-				
Paraphe et code op	érateur : r le responsab	ole:	49 1	oui		non				
Paraphe et code op	érateur : r le responsab	ole:	49 1			-				
Paraphe et code op  Partie à remplir par  Caractérisation for	pérateur : r le responsab melle de tous	ole:	49 1	oui		non				
Paraphe et code op	pérateur : r le responsab melle de tous	ole:	49 1	oui		non				
Paraphe et code op  Partie à remplir par  Caractérisation for	pérateur : r le responsab melle de tous	ole:	49 1	oui		non				
Paraphe et code op  Partie à remplir par  Caractérisation for	pérateur : r le responsab melle de tous	ole:	49 1	oui		non				